Audit of Data Quality
April 2011 Sampling Event
Data associated with "Ground-Water Investigation in Pavillion, Wyoming," QA ID #G-14478
analyzed at US EPA Region VIII Laboratory
ADQ performed by Neptune and Company, Inc.

ADQ Report Date: August 17, 2011.
Four validation Excel spreadsheets are included in this ADQ report and are provided as separate files: April 2011 Pavillion R8 Volatiles Method 8260 Validation Worksheets, April 2011 Pavillion R8 Semivolatiles Method 8270 Validation Worksheets, April 2011 Pavillion R8 TPH DRO Method 8015D Validation Worksheets, and April 2011 Pavillion R8 TPH GRO Method 8015D Validation Worksheets. These worksheets include documentation of the validation process, along with sample and batch information, and recalculations.

1. **Laboratory Data Audited:**

Laboratory (Organization):
US EPA Region VIII Laboratory.

Sample Type(s)/Methods/Analyte(s): Four analytical methods were to be included in this task for the samples identified below: 1) TPH/DRO, 2) 8270 semivolatiles, 3) 8260 volatiles and 4) TPH/GRO.

Sample Identification: EPAMW01, EPAMW02, PGDW5, PGDW20, PGDW30, and PGDW32.
WOs associated with these samples are identified in the support Excel Spreadsheets provided with this Audit of Data Quality Report.

QA Reviewers: Rebecca Shircliff and David Gratson, Neptune and Company, Inc.

Method Information (all four methods provided as separate pdf files):
1) TPH/DRO: EPA Method 8015D (modified), Region VIII Operating Procedure (OP) ORGM-508 r1.0
2) 8270 semivolatiles: EPA Method 8270D (modified), Region VIII OP ORGM-515 r1.1
3) 8260 volatiles: EPA Method 8260, Region VIII OP ORGM-501 Rev 1.1.

4) TPH/GRO: EPA Method 8015D (modified) Purge and Trap, Region VIII OP ORGM-506 r1.0.

File Information: Final Report included in file 1104024,1104026,1104027 final 16 jun 11_S.pdf.

TPH/DRO: Associated Files: April 2011 Region 8 Lab Data Package – LSR 1104024 – Pavillion 2011 1.pdf and April 2011 Region 8 Lab Data Package – Sequence No. 1D26001.pdf

Semivolatiles via EPA Method 8270: Associated Files: April 2011 Region 8 Lab Data Package – LSR 1104024 – Pavillion 2011 1.pdf, April 2011 Region 8 Lab Data Package – Sequence No. 1E05006.pdf, April 2011 Region 8 Lab Data Package – Sequence No. 1E12003.pdf and April 2011 Region 8 Lab Data Package – Sequence No. 1E18003.pdf

Volatiles via EPA Method 8260: Associated Files: April 2011 Region 8 Lab Data Package – LSR 1104024 – Pavillion 2011 1.pdf and April 2011 Region 8 Lab Data Package – Sequence No. 1D29001.pdf

TPH/GRO: Associated Files: April 2011 Region 8 Lab Data Package – LSR 1104024 – Pavillion 2011 1.pdf, April 2011 Region 8 Lab Data Package – Sequence No. 1D25001.pdf

QA/QC Criteria for Analytical Methods: QAPP specified and Laboratory specific QA/QC criteria and limits were used as the basis of this ADQ. Note however, the Pavillion QAPP did not provide specific QA/QC criteria associated with the EPA Region VIII Laboratory methods. The laboratory did provide a QA/QC Summary table (attached as a pdf file entitled R8 Lab Summary QA_QC.pdf). The DoD LCS study refers to a study used to derive statistical control limits for Semivolatile and Volatile analytes in laboratory control samples (spiked blank matrix). The QA/QC Summary table, DoD statistical limits, and information gathered during the TSA at Region VIII (unrelated to this project) were used to evaluate the laboratory against data quality indicators and to assess the usability during this ADQ. Table 1 below is a summary of these QA/QC criteria.

Table 1. Region VIII Laboratory QA/QC Requirements.

QC Type	Semivolatiles (Method 8270D)	DRO (Method 8015D)	GRO (Method 8015D)	Volatiles (Method 8260C)	Frequency
Method Blanks	Preparation Blanks (same as method blank), one with each set of extraction groups	Preparation Blanks (same as method blank), <RL	Instrument Blank (IBL) is the method blank <RL	Method Blank <RL	One per sample set

	within the lab, calibration blanks, <RL				
Surrogate Spikes	"System Monitoring Compounds" use DoD derived limits. concentration 5 ug/mL (20 for tribromophenol) with no dilution. Note, for the Pavillion specific compounds, the surrogate 2-fluorophenol limit is 60-120% in the associated laboratory reports.	60-140% of expected value, o-terphenyl	70-130% of expected value, bromofluorobenzene, added automatically by autosampler	Statistical Limits from DoD LCS Study	Every field and QC sample
Internal Standards Verification.	Every sample, EICP area within ±50% of last ICV or first CCV. Add additional IS if needed for dilutions. (SOP Sections 9.4 and 11.4.6)	NA	NA	EICP area within -50% to +100% of ICAL midpoint standard	Every field and QC sample for applicable methods
Initial multilevel calibration	ICAL: minimum of 6 levels (.25 -12.5 ug/L) , one is at the MRL (0.50 ug/L), prior to sample analysis (not daily) RSD≤20%, r^2 ≥0.990	ICAL: 10-500 ug/L RSD<=20% pr r^2>=0.990	ICAL: .25-12.5 ug/L for gasoline (different range for other compounds) RSD<=20% pr r^2>=0.990	ICAL, RSD<=20% pr r^2>=0.990	As required (not daily if pass ICV)
Initial and	CCV (same source	Daily with each	Daily with each sequence. ICV1	ICV (second source)	CCV At beginning

Continuing Calibration Checks	as ICAL): daily and every 12 hours, 80-120% of expected value	sequence. ICV1 =DRO, ICV2 = surrogate only check 80-120% of expected value	& CCV1 = gasoline, ICV2 & CCV2 = BTEX+MTBE+naphthalene 80-120% of expected value	% R ±20% CCV % R ±20%	of sample set, every tenth sample, and end of sample set
Second Source Standards	ICV1 is from a second source (includes 7 special compounds) Once after each ICAL, 70-130% of expected value	ICV1 is from a second source, 80-120% of expected value	ICVs are from different source. 80-120% of expected value	ICV (second source) % R ±20%	Each time calibration performed
Standard Reference Material (SRM)	Once per batch, limits based on SRM certification	See below	See below	NA	
Laboratory Control Samples (LCS)	Blank Spike, one per extraction group included once per sequence or every 20 samples. 1mL into 1 L of sample at mid level. Statistical Limits from DoD LCS Study (rounded to 0 or 5)	Often use SRM as LCS, if so limits based on certification information, otherwise 70-130% of expected value	Often use SRM as LCS, if so limits based on certification information, otherwise 70-130% of expected value.	Spike Recovery within Statistical Limits from DoD LCS Study	One per analytical batch or every 20 samples, whichever is greater
Matrix Spikes (MS)	Same as LCS	Same as LCS (70-130%, may develop statistical based in future)	Spike with ICAL mix Gasoline 70-130%, others DoD limits	Spike Recovery within Statistical Limits from DoD LCS Study	One per sample set or every 20 samples, whichever is more frequent
MS/MSD	Once per batch or every 20 samples. RPD $\leq$ 20% Note, the limits in the Reg VIII lab files is $\leq$ 30%	RPD $\leq$ 25	RPD $\leq$ 25	RPD$\leq$30%	One per sample set or every 20 samples, whichever is more frequent

Detection Limit Standard (CRL)	run MDL study approximately annually	DL= RL, ICAL run down to 10 ug/L MDL study approximately annually	DL= RL, MDL study approximately annually	±50% of expected value	CRLs not routinely analyzed, only report to RL (lowest standard of cal model)
Reporting Limits*	0.1 µg/L (generally)[1]	20 µg/L[1]	Gasoline is 20 µg/L[2] Other compounds RL is from 1-200, compound specific	Not specified in QAPP, as EPA RSK was doing the analysis for Killdeer	NA (part of ICAL)
Other Method Specific	GC/MS tuning (DFTPP) : prior to ICAL and at beginning of each 12-hour period.			GC/MS tuning (BFB): prior to ICAL and at beginning of each 12-hour period.	

[1]Based on 1000 mL sample to 1 mL extract
[2]Based on a 5 mL purge
*these limits are compound dependent (see table below)

2. Summary of Assessment

In cases where QA/QC issues were identified, the samples had been properly qualified by the laboratory in the final report.

Observations

1. **Recalculations do not match reported (see spreadsheets)**

    a. The values for four 8260 VOC compounds (acetone, MIBK, 2-hexanone, and m/p-xylenes) varied from the reported values. MIBK is within 10% of the reported value; however m/p xylenes, 2-hexanone, and acetone exceeded the reported value by more than 10%. The differences are likely due to the use of quadratic or linear calibration models versus the use of average response factors. Toluene quantification was based upon the average response factor and this value was reproduced. The laboratory has been contacted to identify the constants used in the calibration models in order to reproduce the concentration values.

    b. The values for two 8270 SVOCs compounds (benzoic acid, and bis [2-ethylhexyl] phthalate) also varied from the

reported values by greater than 10%. The differences are likely due to the use of quadratic or linear calibration models versus the use of average response factors.

The values that were recalculated during the assessment were equal to the laboratory reported values, when the basis of the quantification by the laboratory, and this assessment were the average response value. Differences between reported and recalculated values are noted for those reported values in which the laboratory used a linear or quadratic model.

2. **Holding Times for VOC Samples.** Holding times were not met for all samples, see Question 8 below, missing the deadline by 1-2 days. See the VOC worksheet. The associated samples were qualified by the laboratory and identified in the final report case narrative.

Editorial Comments

1. **DRO Analysis Method.** The results report for DROs lists 8015**B** as the analysis method, see Question 6 below in table. This should be 8015D.

2. **EPA Tag No. for GRO.** The results report for GROs does not use a consistent EPA Tag No. Instead it alternates between BTEX/GAS and BTEX/GRO.

QA issues based on Field QA/QC: The VOC (Method 8260) Field Blank (Lab Number 1104024-08) collected on 4/18/2011 had the compounds at the following concentration, with reporting limits in parenthesis; all units are µg/L:

2-butanone 0.640 (0.500), 2-hexanone 0.290 (0.250), acetone 1.03 (1.00), m&p xylene 0.690 (0.500) and methacrylonitrile 0.270 (0.250).

The native (field) samples associated with this Field Blank should be evaluated for these same analytes.

ITEMS REVIEWED

Number	ADQ Issue	Yes	No	NA	Comments
File Information					
1	Provide File names: See Section 2.0 above.				
Sample Information					
2	Are samples uniquely identified and correctly transcribed throughout the data package to the summary of results?	**X**			Samples are uniquely labeled as EPAMW01, EPAMW02, PGDW5, PGDW20, PGDW30, and PGDW32 for all methods. In addition, samples are identified by unique Lab IDs throughout the raw data packages for all methods.
3	Does sample collection documentation indicate that samples were collected as described in the QAPP, and the schedule and volumes in the planning documentation?	**X**			The only sample collection documentation within the report files is: date/time sample was collected, sample volume and pH for DROs and number of samples collected. Any additional specific sampling information is not expected to be in the laboratory report. So, this is acceptable.
4	Does sample collection documentation indicate appropriate preservation?	**X Partial**			According to the Pavillion QAPP, none of the samples for Reg VIII were to be acidified in the field. DRO samples were acidified upon receipt at the lab for analysis. All samples were preserved on ice during shipment. There is no clear indication of how the samples were preserved after receipt by the labs (e.g. temperature stored at).
5	If applicable, is chain-of-custody documentation complete? (Contains relinquished and received	**X**			COC documentation was found in files associated with specific work orders/batches.